

## Accepted Manuscript

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PII: S0261-3069(15)00101-6

DOI: <http://dx.doi.org/10.1016/j.matdes.2015.03.018>

Reference: JMAD 7134

To appear in: *Materials and Design*

Received Date: 11 October 2014

Revised Date: 10 March 2015

Accepted Date: 12 March 2015



Please cite this article as: Han, Y., Li, J., Huang, G., Lv, Y., Shao, X., Lu, W., Zhang, D., Effect of ECAP numbers on microstructure and properties of titanium matrix composite, *Materials and Design* (2015), doi: <http://dx.doi.org/10.1016/j.matdes.2015.03.018>

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## Effect of ECAP numbers on microstructure and properties of titanium matrix composite

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### Abstract:

In this study, equal channel angular pressing (ECAP) of (TiB+TiC)/Ti6Al4V titanium matrix composite was successfully carried out at 800°C, the effect of pass number on the microstructure and mechanical properties was investigated. After each pass of ECAP, the evolution of the microstructure and mechanical properties of (TiB+TiC)/Ti6Al4V composite during thermal-mechanical processing was studied, the Vickers micro-hardness measurements and tensile testing were performed at room temperature. The results showed that the size of the reinforcements and grains were both fully refined to a smaller scale, a number of more homogeneous TiB short fibers and TiC particles have been attained after four ECAP passes. The tensile strength increased with increasing ECAP numbers and saturated after four ECAP passes to a yield strength of 1200MPa, and microhardness was also significantly improved by means of this processing technology, the ductility of titanium matrix composite after the four ECAP passes was slightly greater than the first ECAP pass.

**Keywords:** Titanium matrix composite, ECAP, Mechanical property, Microstructure.

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## 1. Introduction

Mirzakhani et al [1] pointed that severe plastic deformation (SPD) was one of the best methods to promote profound changes on the microstructure and properties of metals and alloys. Most investigations [2-3] developed equal channel angular pressing (ECAP) as one of the most feasible severe plastic deformation (SPD) techniques for industry manufacturing. Shaeri et al [4] reported that as a typical SPD technology, the main advantage of this method was that it can produce ultra-fine grained (UFG) structure and improve properties effectively. Up to now, most of the investigations related to ECAP on titanium and its alloys have been successfully confirmed to process samples especially CP-Ti, Ti6Al4V, metastable  $\beta$  titanium alloys and Ti-Ni alloys. Kang, et al [5] studied the mechanical behavior and microstructure evolution of CP-Ti in enhanced multi-pass ECAP and cold extrusion, the results showed that the ultimate tensile strength of CP-Ti increased remarkably from 295.5MPa to 791.9 MPa with significant refinement of the microstructure. Ko, et al [6] investigated the effect of initial microstructure on the formability of Ti6Al4V alloy, their work reported that most  $\alpha$  and  $\beta$  grains were significantly refined to 0.2-0.3  $\mu\text{m}$  in diameter consisting of high angle grain boundaries, the tensile strengths were extremely improved by the ECAP process. However, these previous works focused on the pure Ti and titanium alloys. There are few reports on ECAP of titanium matrix composite with higher strength.

Considering the industrial importance of titanium matrix composite, it is essential to establish an optimum processing technology for such difficult-to-fabricate materials. But the incorporation of high modulus and high strength reinforcements into titanium, it makes titanium matrix composite possessing high specific strength but low ductility at room and elevated temperature. Moreover, Lu, et al [7] investigated the reinforcements usually showed different shapes such as short-fiber shape, dendritic shape and equiaxed or near equiaxed shape by using common casting technology. Sun, et al [8] reported that these reinforcements were on a large range of 2~50 $\mu\text{m}$ , which dispersed in a homogeneous manner in the composite. To solve this problem, it is useful to use a severe plastic deformation technology to homogenize the reinforcements in the composite. ECAP has a high

hydrostatic pressure and shear strain, which can refine the grains and make the reinforcements distribute homogeneously [9]. Hence, it is of interest to apply ECAP process for fabricating the titanium matrix composite.

According to the current literature, extensive studies have pointed out that TiB short fibers and TiC particles are the best reinforcements for titanium matrix composite [10]. The composite reinforced by TiB fibers and TiC particles had higher mechanical properties than the composite reinforced by only TiB fibers or only TiC particles, respectively [11~13]. In this study, the Ti6Al4V alloy is used as matrix alloy of the composite for its good mechanical properties and wide applicability in the industries, the composite is manufactured from the in situ chemical reaction between Ti6Al4V and B<sub>4</sub>C addition, and the microstructure and mechanical properties with increasing the number of ECAP passes were investigated for (TiB+TiC)/Ti6Al4V composite. Although a considerable amount of work had shown that Ti6Al4V alloy can achieve a better balance between high strength and good ductility utilizing the ECAP technique [14]. Rare literatures have been focused on the titanium matrix composite processed by ECAP. Therefore, it is extremely useful to investigate the variation of microstructure and mechanical properties of ECAP processed (TiB+TiC)/Ti6Al4V composite.

## 2. Experimental material and procedures

### 2.1 Sample preparation

In this work, the studied material (TiB+TiC)/Ti6Al4V composites were fabricated into ingots with the size of  $\Phi 120\text{mm}$  by the consumable vacuum arc-remelting furnace. The addition of small amount of B<sub>4</sub>C to Ti produced TiB and TiC during melting process by the in situ chemical reaction:  $5\text{Ti} + \text{B}_4\text{C} \rightarrow 4\text{TiB} + \text{TiC}$ , and then the ingot was hot forged into a billet 25 mm in diameter and 1000mm in length, followed by annealing treatment at 600°C for 30 minutes and air cooling, respectively. Finally, the (TiB+TiC)/Ti6Al4V composite was cut into cubic specimens of 10mm×10mm×140mm for ECAP processing. Fig. 1 shows the optical micrograph of the initially forged samples before ECAP pressing. It is observed that the microstructure of specimen shows equiaxed structure and the volume fraction

of the microstructure consisted of approximately 85% equiaxed  $\alpha$  phase, 10% long TiB fibers and large TiC particles. The size of the equiaxed  $\alpha$  phase is about 8~10 $\mu$ m in diameter, TiB fibers is about 20 $\mu$ m in length and width of 5 $\mu$ m, TiC particle is about 15 $\mu$ m in diameter with an irregular shape.

## 2.2 ECAP processing

Special die-set with enlarged angles of channel intersection equal to 120° was fabricated to process ECAP alloys. The route Bc was applied, a billet was turned clockwise along its longitudinal axis by 90° after each pass. The pressing temperature was 800°C. A schematic illustration of the ECAP process was shown in Fig.2. In order to investigate the effect of the amount of deformation and pass numbers on the microstructure and properties of (TiB+TiC)/Ti6Al4V composite. For each pass, the ECAP die was heated to 550°C, and the cubic specimen was held for 20min to reach the temperature stabilization. Before inserting into the ECAP die channel, the sample was coated with graphite paste to assure lubrication during ECAP process. After that, the sample was pressed at the speed of 8mm/min, and the samples on each processing were extruded for one, two, three and four passes at the same temperature. The lubricant of graphite paste was also applied to reduce the friction between the die and specimen during the ECAP process. After each ECAP process, the sample should be quickly removed and water quenched to keep the microstructure. Longitudinal sections of the samples were prepared for optical microscopy (OM) and scanning electron microscopy (SEM) analyses.

## 2.3 Microstructure and property tests

To observe the microstructural evolution, the deformed specimens were sectioned parallel to compression axis from one side of the deformation specimens, and the cut surface of the smaller part was prepared for metallographic examination by means of optical metallographic (OM) and scanning electron microscopy (SEM). The polished samples were etched with a solution of 30% HNO<sub>3</sub>+10% HF in water for 5 seconds. Tensile tests were performed at room temperature using a universal testing machine Zwick Z100/SN3A. Flat samples with a gage length of 18mm and a cross-section of 3mm

$\times 1.6\text{mm}$  were wire Electrical Discharge Machining (EDM) cut from the titanium matrix composite billets. A strain rate of  $10^{-3}\text{ mm/s}$  was used for the all tests. Yield strength and elongation to failure at the necking cross-section were measured. The Vickers microhardness (HV) was measured on polished surface. A load of 5kgf was applied for a dwell time of 10s. Ten measurements were made for each sample, and all reported microhardness values represented the average of the measurements.

### 3. Result and discussion

#### 3.1 Phase identification and microstructure

Fig.3 shows X-ray diffraction patterns for initial and ECAP-processed samples. They all indicated that the phases present are  $\alpha$ -Ti,  $\beta$ -Ti, TiB and TiC. The results of X-ray diffraction analysis confirms that the (TiB+TiC)/Ti6Al4V titanium matrix composite could be fabricated by common casting technique using chemical reaction between titanium matrix metal and  $\text{B}_4\text{C}$  powder. Compare with different ECAP passes, there is no significant difference in the X-ray diffraction patterns. Fig. 4 shows the optical micrographs illustrating the microstructure evolution of the (TiC+TiB)/Ti6Al4V composite with ECAP deformation. The matrix alloy exhibits a near-fully equiaxed microstructure. After hot ECAP extrusion processing at first pass (Fig.4a, 4e), the microstructure consists of elongated equiaxed  $\alpha$  phases in the direction of extruding with average grain size estimated as about  $20\mu\text{m}$  in length. TiB short fibers in titanium matrix composite show good alignment along the extrusion direction, and the TiC large particles disperse in the specimens with diameter of about  $14\mu\text{m}$ . After one pass, it can be seen that the equiaxed  $\alpha$  phase of the matrix alloy still keep the initial size, and the reinforcement's distribution is inhomogeneous. Furthermore, many TiC particles and TiB fibers are clustering in small zones, and some TiC particles with pores are observed in the ECAP processed sample. According to the principle of ECAP, ECAP could induce larger shear strain into samples [15]. The TiB short fibers and TiC particles are smaller and the homogeneity of the reinforcement's distribution is improved. With the deformation proceeding, after four passes, the reinforcements are refined and rearranged significantly during ECAP processing. This owns to the continuous shearing in Bc route makes the reinforcements distribute homogeneous. As shown in the

Fig.4d, 4h), 4-pass processed sample, the microstructure of the matrix alloy also become more uniform, with the average grain size estimated as about 14 $\mu$ m in diameter. It seems that the grain size of the matrix alloy has a little change due to the generation of dynamic recovery and dislocation, which owns to the relative high extruding temperature [16].

From optical micrographs' observations, large volume fraction reinforcements show very inhomogeneous particle distribution, which could limit the formability of the titanium matrix composite. So, it is interesting to investigate the distribution and morphology of reinforcements since they have a strong effect on the deformation of titanium matrix composite. Fig. 5 shows the SEM micrographs of (TiB+TiC)/Ti6Al4V composite after ECAP for different passes. After the first ECAP process, the initial reinforcements have a little change comparing with the original microstructure. It owns to the employed dies with channels making 120°, there is a lower strain existed in the first pass, see Iwahashi's formula [15]. But for the second pass, the TiB fibers are severely fragmented and TiC particles are broken heavily. The evolution of TiB fibers and TiC particles are investigated by measuring the aspect ratio and particles length using image analysis software. The aspect ratio is defined as the ratio of maximum and the minimum lengths of the rectangle with the smallest area that can be draw around the particle. It shows that the aspect ratio of TiB fibers and TiC particles in all specimens decrease with increasing the number of ECAP passes. Simultaneously, the length of TiB fibers decreases when the billet is deformed by ECAP. The results are in accordance with the results of previous studies on processing for particle reinforced Al composites [17]. Regardless of the different number of ECAP passes in preparing (TiB+TiC)/Ti6Al4V composite, the average aspect ratio of TiC particles is similar at four passes. However, the reduction in TiC particles size is significant different. Refined TiC particles could be obtained significantly with the increasing number of ECAP passes.

### 3.2 Mechanical property

Tensile test curves for the number of ECAP processed billets were plotted in Fig. 6. The mechanical properties were obtained by the methods mentioned in section 2. 3. It could be seen clearly that the

influence of the number of passes on mechanical property is obvious. After the first pass, the tensile strength of the ECAP processed billets increases significantly to 1100MPa, while the elongation is only 2.7%. Then the plasticity is improved as the number of pass increasing, the tensile strength increases up to the biggest value (1205MPa) in the third ECAP pass. Comparing with other materials which are subjected to rolling deformation, the strength should increase while the plasticity decreases in a certain amount. But for the ECAP processed titanium matrix composite, the plasticity is improved with the increase of pass number. The reason of the improvement is attributing to the phase boundaries increasing resulted from the refinement of reinforcement and the grain size after one ECAP pass number [18]. The effect of work hardening behavior becomes obvious gradually after three passes (Fig. 6), while the tensile strength decreases slightly after four passes. The decrease of strength and the increase of ductility could be the interaction of refinement and working hardening [19]. During the third ECAP pass, the refinement of TiB fibers and TiC particles would enhance the dislocations, which results in dislocation density increasing quickly in the boundary of the refinements, and the work hardening phenomenon is serious. After the four passes, the ductility increases from 3.8% to 4.5%, it is attributed to the annihilation of the dislocations [20]. As a result, (TiB+TiC)/Ti6Al4V composite processed exhibits higher elongation but similar strength compared with the sample ECAP processed after the first pass.

From above results, it is clear that the mechanical behavior of different ECAP passes differs from each other. The difference in the ductility levels can be attributed to their reinforcements' difference and also slight variations in the strength. Although their ECAP procedure is quite similar, resulting in the microstructure of matrix metal is similar for each pass, this shows the effectiveness of reinforcements in achieving smaller grain sizes in titanium matrix composite. Fig.7 shows the SEM images of titanium matrix composite near fracture surfaces after room temperature tensile test. It can be seen that the reinforcements play an important role on the tensile properties of the composite. In the first ECAP pass (Fig. 7a, 7e), some long TiB fibers (with high aspect ratio) near fracture surface



fractured, which indicates that the TiB fibers are bearing the tensile stress in the room tensile tests. The fracture mechanism was quite typical for titanium composite at room tensile test [21]. With the number of ECAP passes increasing (Fig. 7b, 7f), some large TiC particles severely break with an angle of about  $60^\circ$  along the tensile direction. There is an interfacial debonding behavior happened between the TiB short fibers and the matrix alloy, which is observed in Fig. 7g. According to the study by Bowen, et al [22], large shear strain was drawn in the ECAP process. After three ECAP passes, the TiB fibers and TiC particles size are decreasing heavily, interfacial debonding is easy to take place in the two ends of the reinforcement, which is due to a concentration of stress in the end of the reinforcement with the increase of the shear strain. Furthermore, lots of cavities will immediately generate around the TiB fibers and TiC particles. It could be one of the dominant reasons for the failure of the sample extruded in the next ECAP pass. Fig. 7d and Fig. 7h show the image of the sample processed after 4 pass. Some small cracks could be observed around the TiB short fibers, and a few broken TiC particles are gathering together. It is found that the reinforcements' load-bearing effect would be less dominant than the dislocation density effect for the refinement of the reinforcement dispersion strengthening, which is accordance with the observation in Fig. 7.

The Vickers microhardness (HV) is measured on the initial and ECAP-processed billets, which are shown in Fig. 8. Each microhardness value is determined as the average of eight measurements after getting rid of the maximum and minimum measurement. It is evident that the microhardness increases gradually with the increasing of ECAP pass number, and the lowest hardness is obtained in the initial sample without ECAP processing, while the highest average value is measured for sample after 4 pass. As discussed before, it is well known that the reinforcements disperse refinement and dislocation density have a significant effect on strength. When the sample is extruded for the first ECAP pass, the work hardening behavior indicates that the dislocation density will have a sharp rise, which leads the enhancement of the microhardness. Moreover, by increasing the number of ECAP passes, the refinement of the reinforcement and the grain tend to acquire a small aspect ratio and high fraction of

high-angle boundaries. Sordi and Ferrante [23] believed these features led to strength enhancement, while the microhardness is a little affected. Therefore, the improvement in the microhardness is mostly due to the ECAP extrusion number, which means that the size of reinforcements and grains in the (TiB+TiC)/Ti6Al4V composite processed by ECAP play a dominant role in increasing the micro-hardness.

### 3.3 Analysis of fracture surfaces

Fracture surface of specimens by different ECAP passes, which had been tested under the tensile test. Fig.9 shows the fracture surface of the ECAP-processed (TiB+TiC)/Ti6Al4V composite. It is important to note that only ductile failure and typical dimple failure morphology under the tensile test have been observed for all ECAP passes. Two distinct-types dimples are observed: (1) the region of the edge of the sample reveals shallow near-equiaxed shaped dimples, and (2) some big and deep dimples are observed surrounding the discontinuous TiB short fibers and TiC particles. Moreover, a population of voids of varying size and fine microscopic cracks exist in the fracture surface. In most instances, the small cracks could pass through the reinforcements and fracture them. As shown in Fig. 9b and Fig.9c, some cracked TiC particles and TiB short fibers can be observed. It indicates that the interfacial cohesion between the titanium matrix alloy and the reinforcements is strong enough and the reinforcements can undergo load and improve the strength and ductility. In Fig. 9d, the submicrometer-sized dimples are found covering the edge and center fracture surface are indicative of good ductility of the titanium composite, which can be an explanation of the highest fracture strain of the sample pressed after four ECAP passes.

## 4. Conclusion

To sum up, due to the refinement of TiB fibers and TiC particles during the ECAP pressed in the titanium matrix composite, the improvement in uniformity of the reinforcements finally causes a positive effect on the tensile strength of the titanium matrix composite. The enhancement of ductility is dominated by the decreasing of reinforcements' size, and the homogeneity of the reinforcements'

distribution through four passes is better than that through one pass. The shear strain directly influences the distribution of TiB short fibers and TiC particles. Moreover, the microhardness is affected mostly by the ECAP extrusion numbers. With the increasing of the extrusion numbers, the small size reinforcements tend to acquire high dislocation density surrounding the TiB fibers and TiC particles, which leads to an enhancement of the microhardness. In addition, the ductile fracture mode can be deduced for the titanium matrix composite due to two distinct-types dimples which are observed in the fracture surface.

### Acknowledgments

The authors are grateful to the National Nature Science Foundation of China (Grant No: 51371114), the 973 Program under Grant No: 2012CB619600, the China Postdoctoral Science Foundation (Grant No. 2014M550235), the Shanghai Postdoctoral Sustentation Fund (Grant No. 14R21410900 ) and Shanghai Academy of Spaceflight-Joint Research Centre of Shanghai Jiao Tong University advanced aerospace technology (No. US-CAST2012-14)

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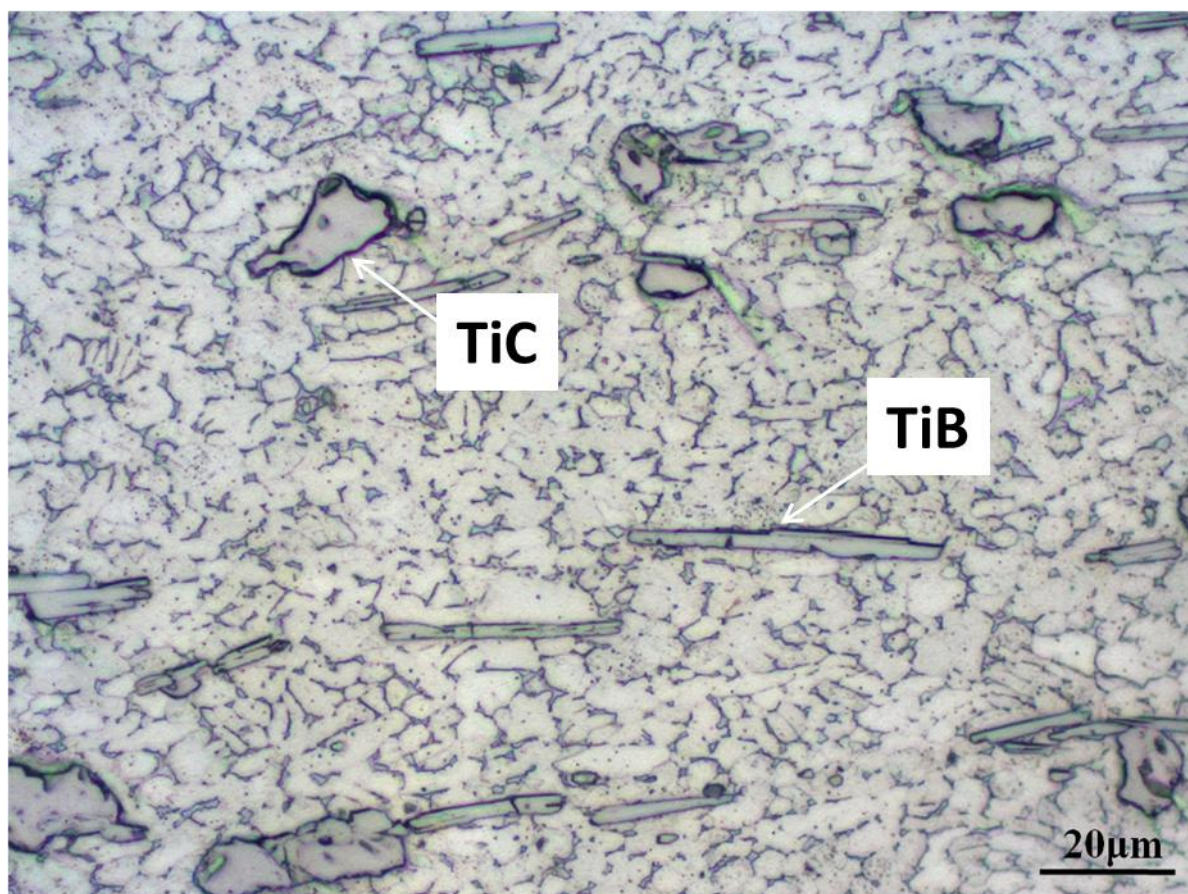


Fig.1 Microstructure of the initial titanium matrix composite billet

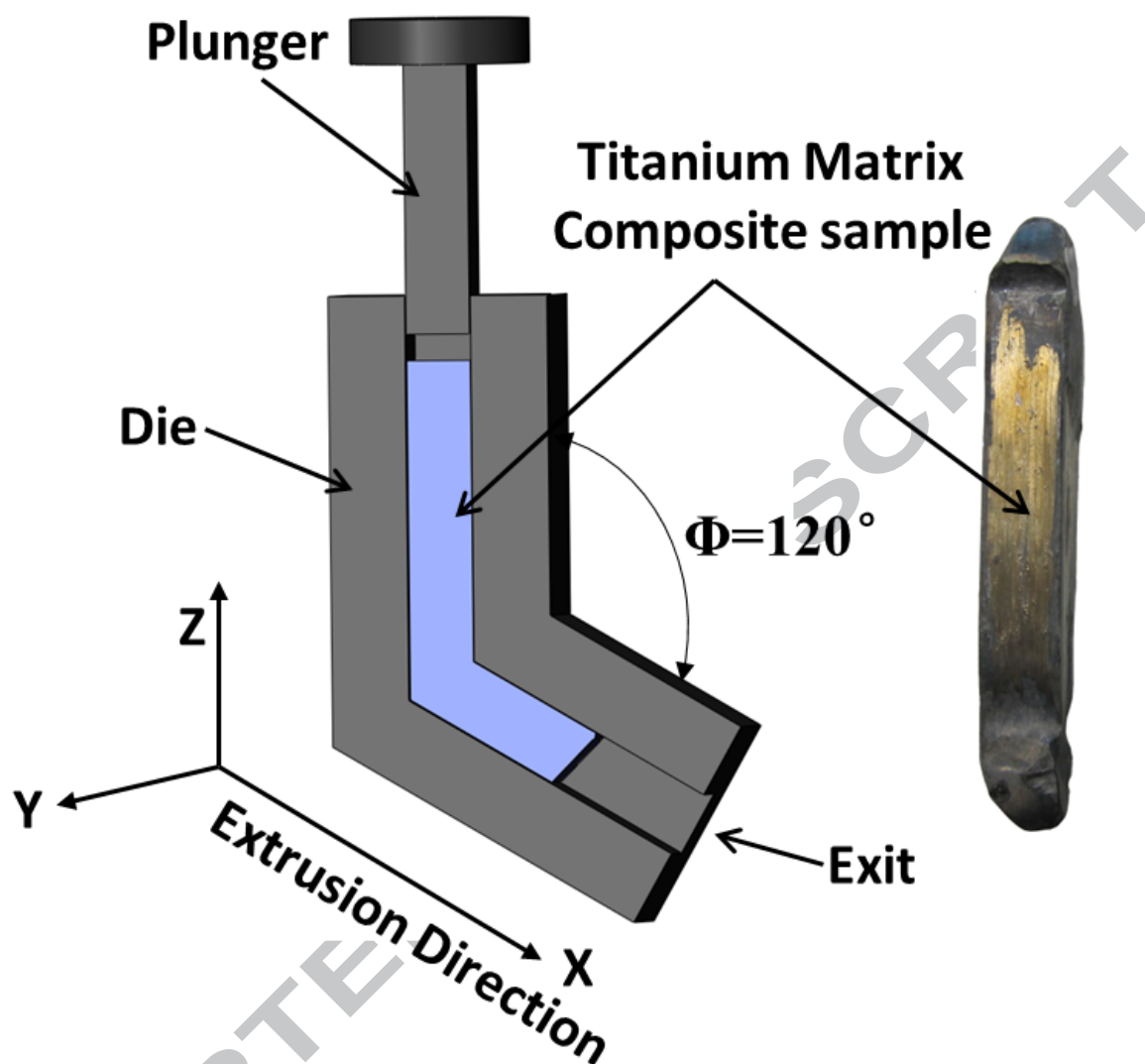


Fig.2 Schematic diagram showing the continuous ECAP process



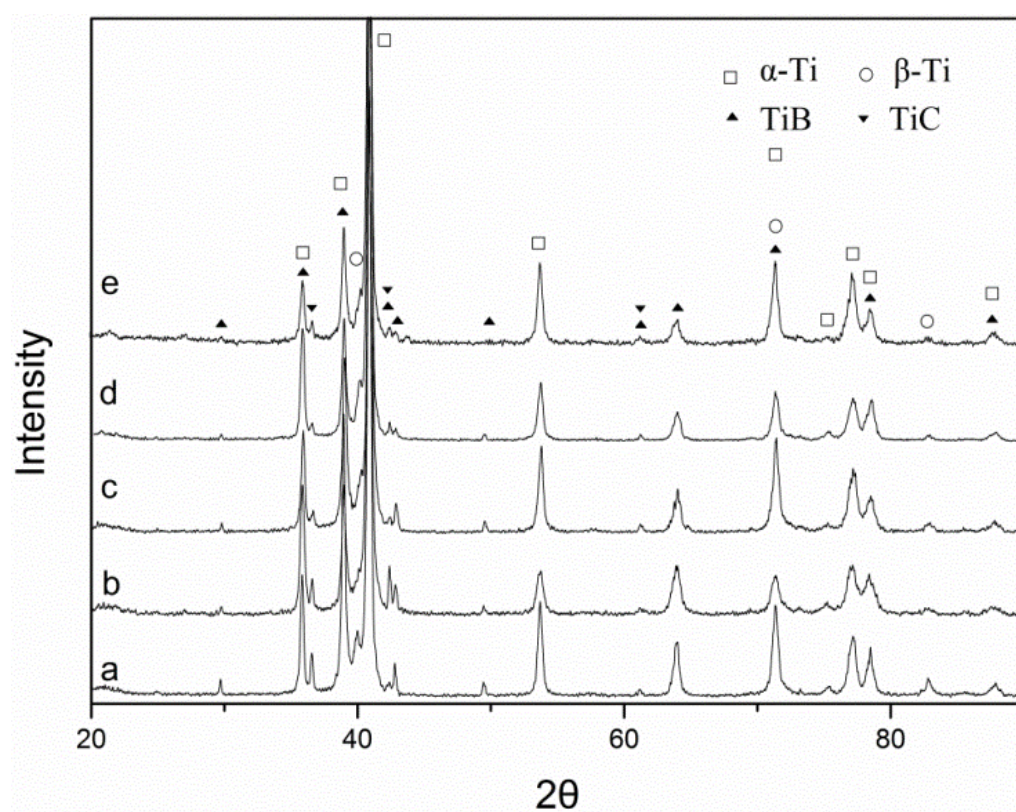


Fig.3 X-ray diffraction patterns (a) Initial material, (b) 1pass (c) 2 passes (d) 3 passes (e) 4 passes

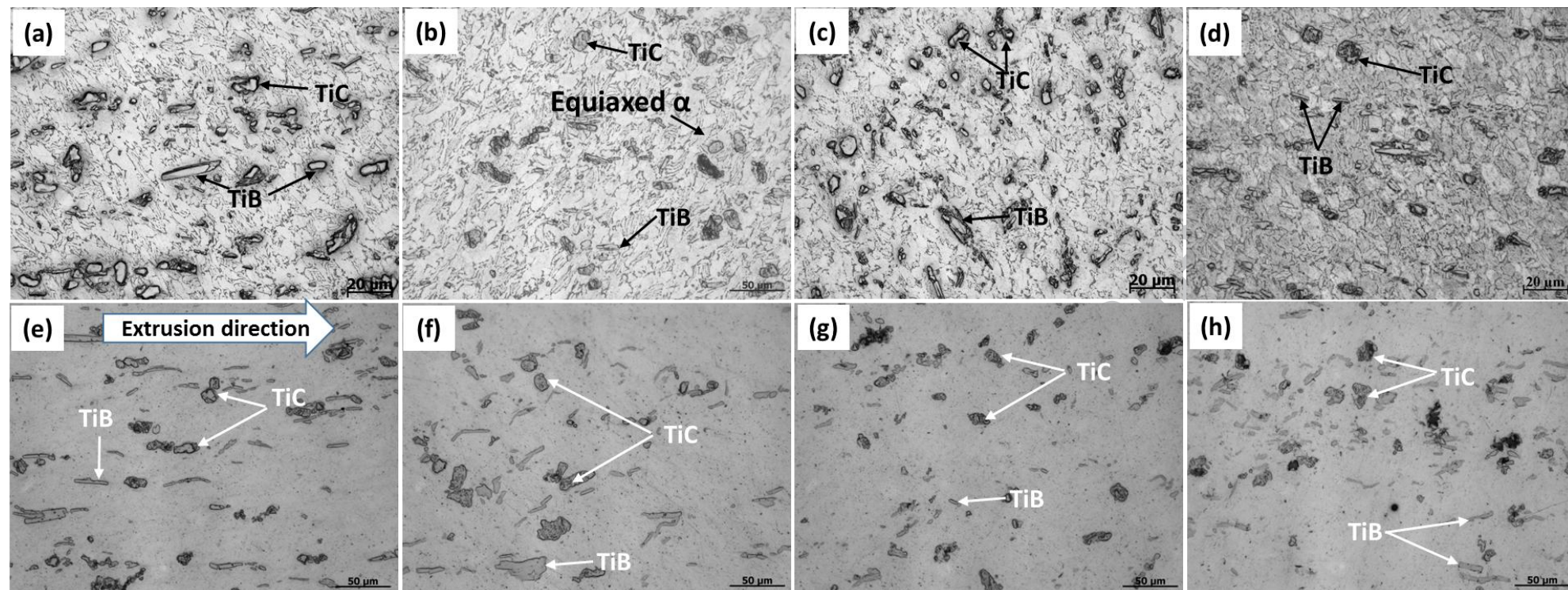


Fig.4 Optical microscope images of the (TiB+TiC)/Ti6Al4V composite

(a, e) First pass, (b, f) Second pass (c, g) Third pass (d, h) Forth pass



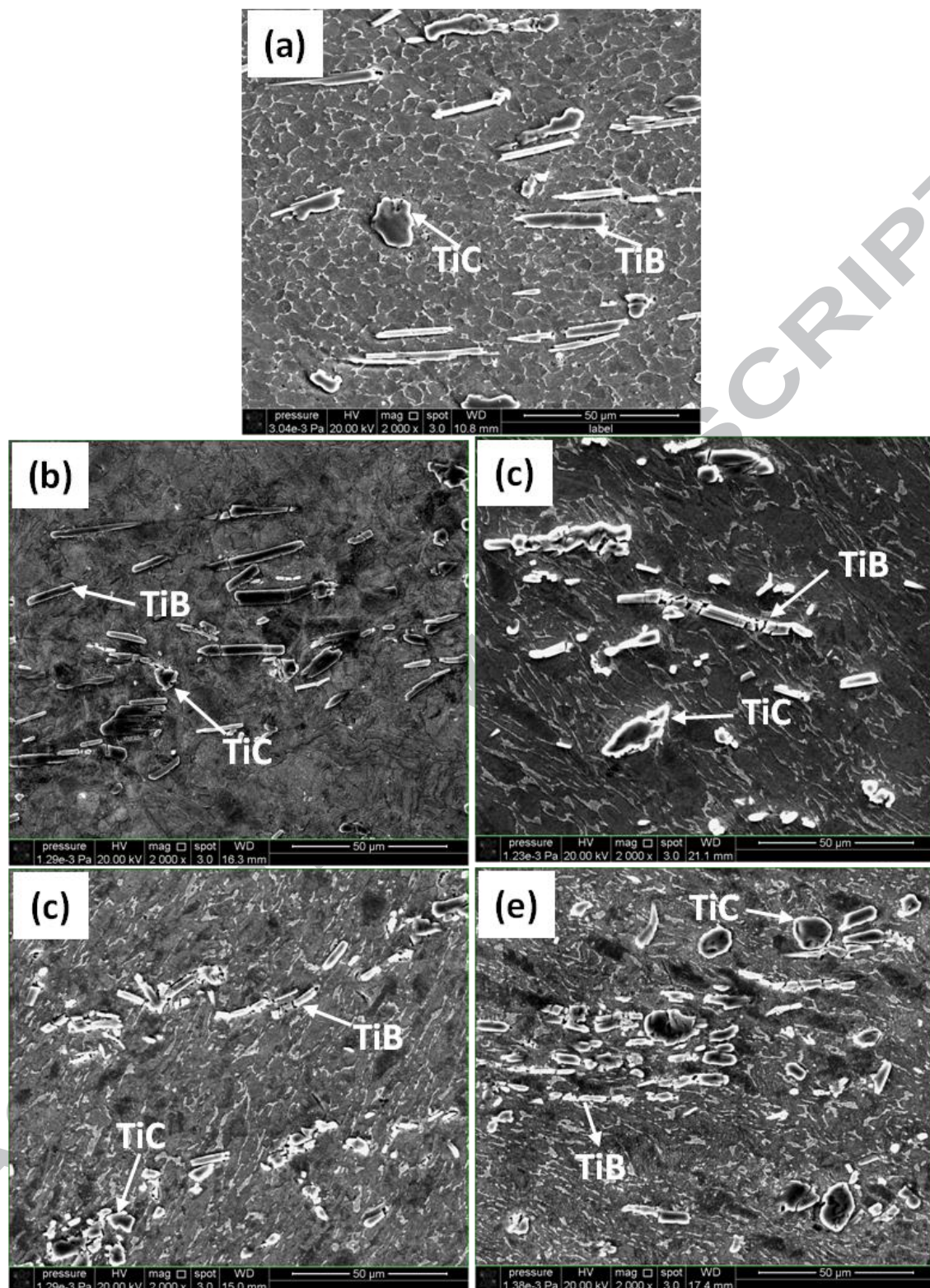


Fig.5 The evolution of the reinforcements in the ECAP processed (TiB+TiC)/Ti6Al4V composite

(a) Original microstructure (b) First pass, (c) Second pass (d) Third pass (e) Forth pass.

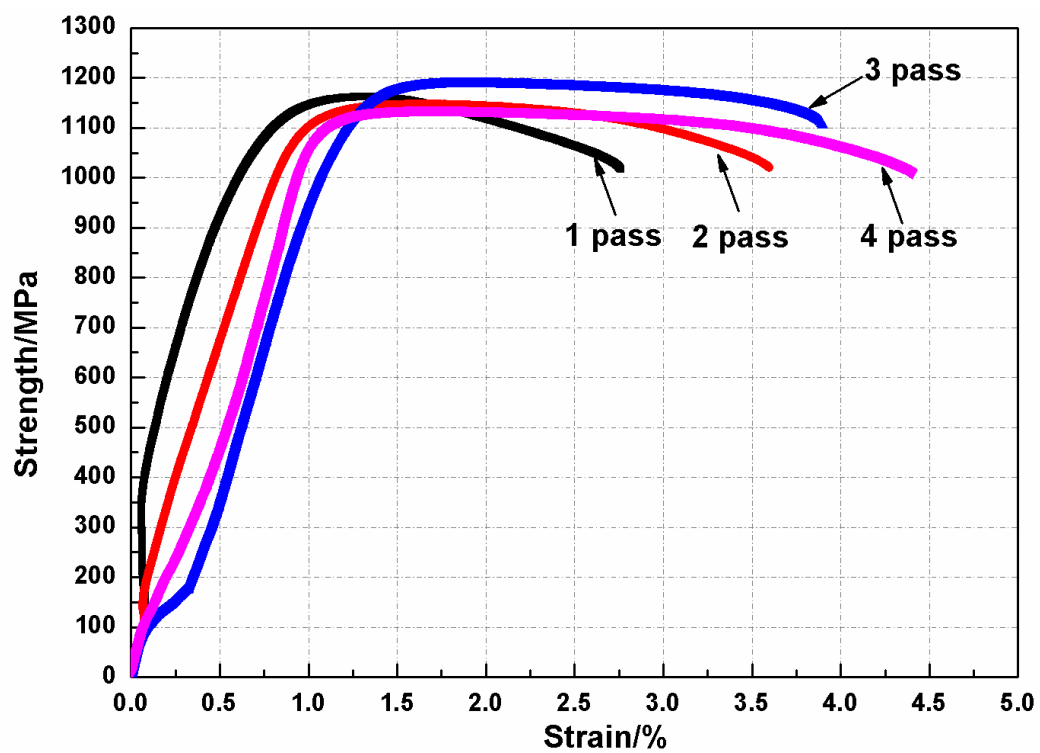


Fig. 6 Tensile test curves for the ECAP processed billets for different passes

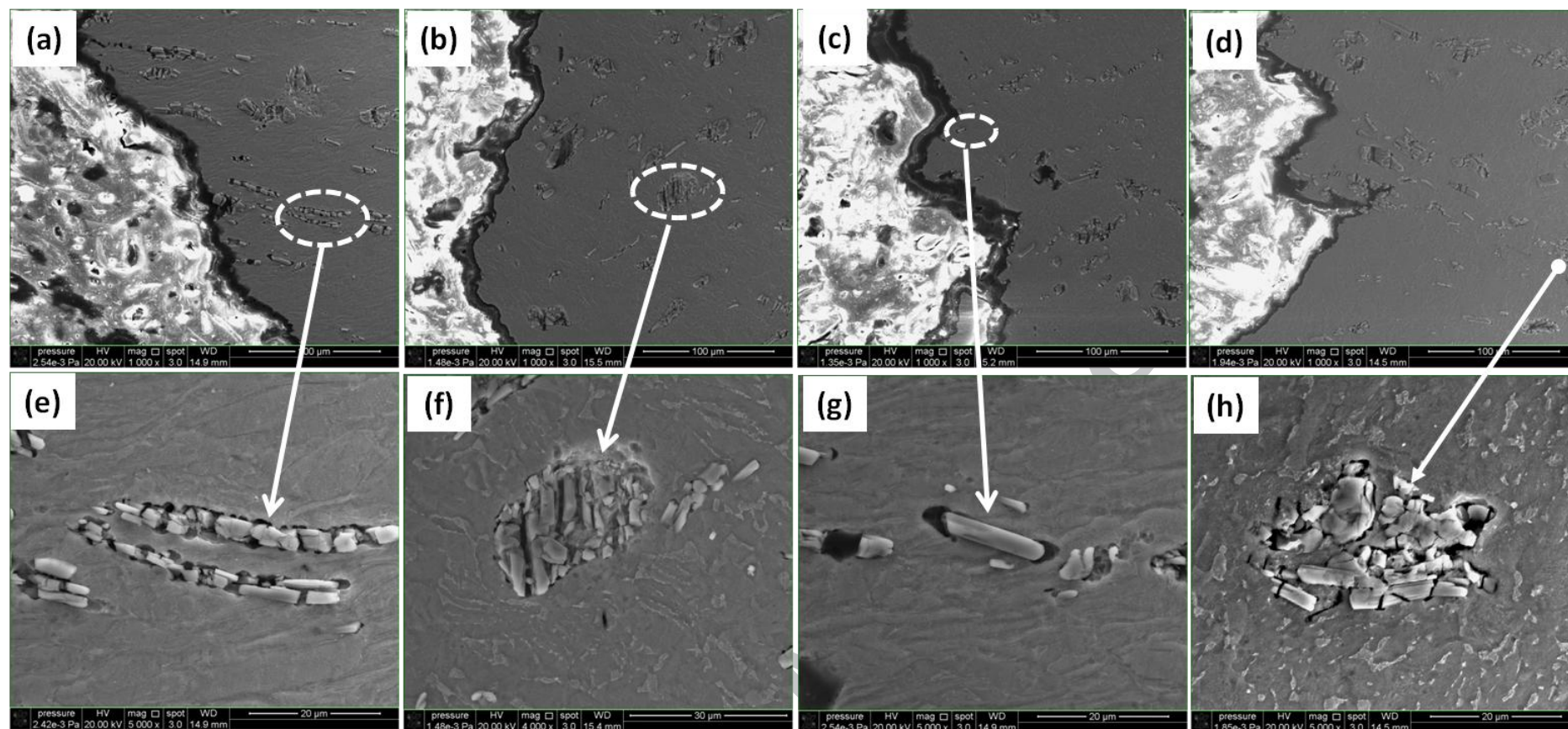


Fig. 7. SEM micrographs of the tensile samples along the load sections near the fracture surfaces

(a, e) 1 pass, (b, f) 2 passes (c, g) 3 passes (d, h) 4 passes



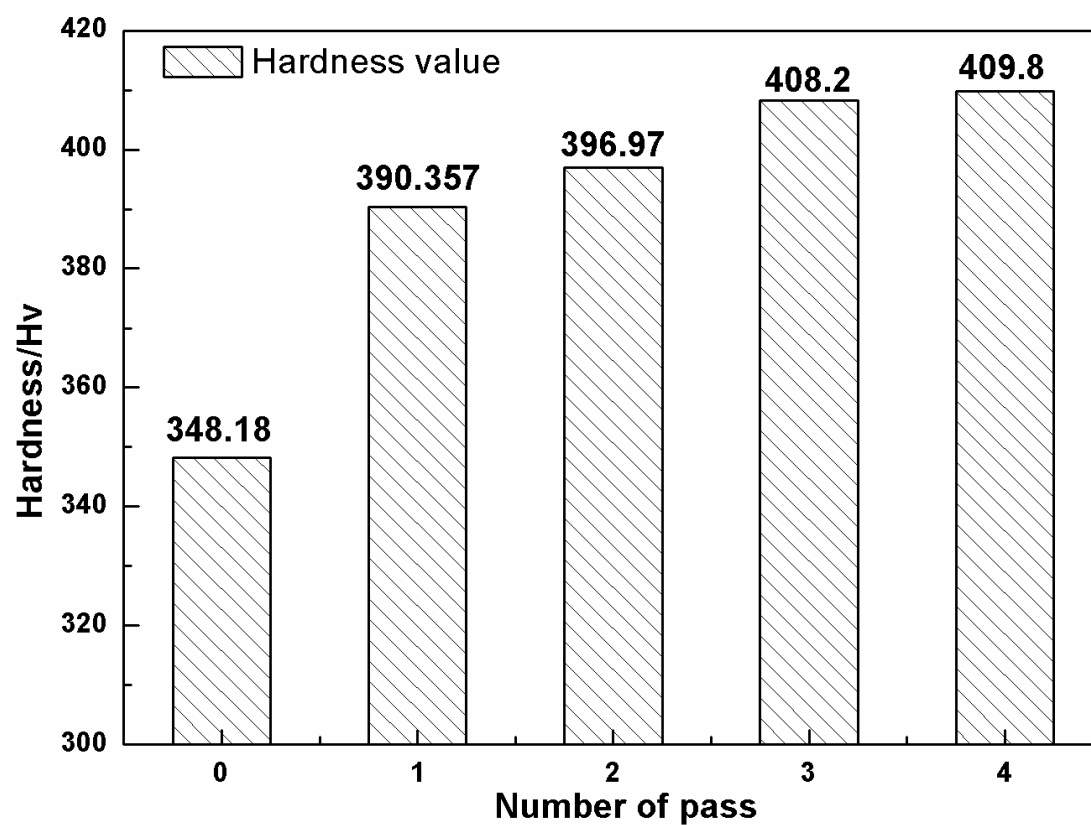


Fig.8 The microhardness of (TiB+TiC)/Ti6Al4V composite varying as ECAP number of pass in the longitudinal section.

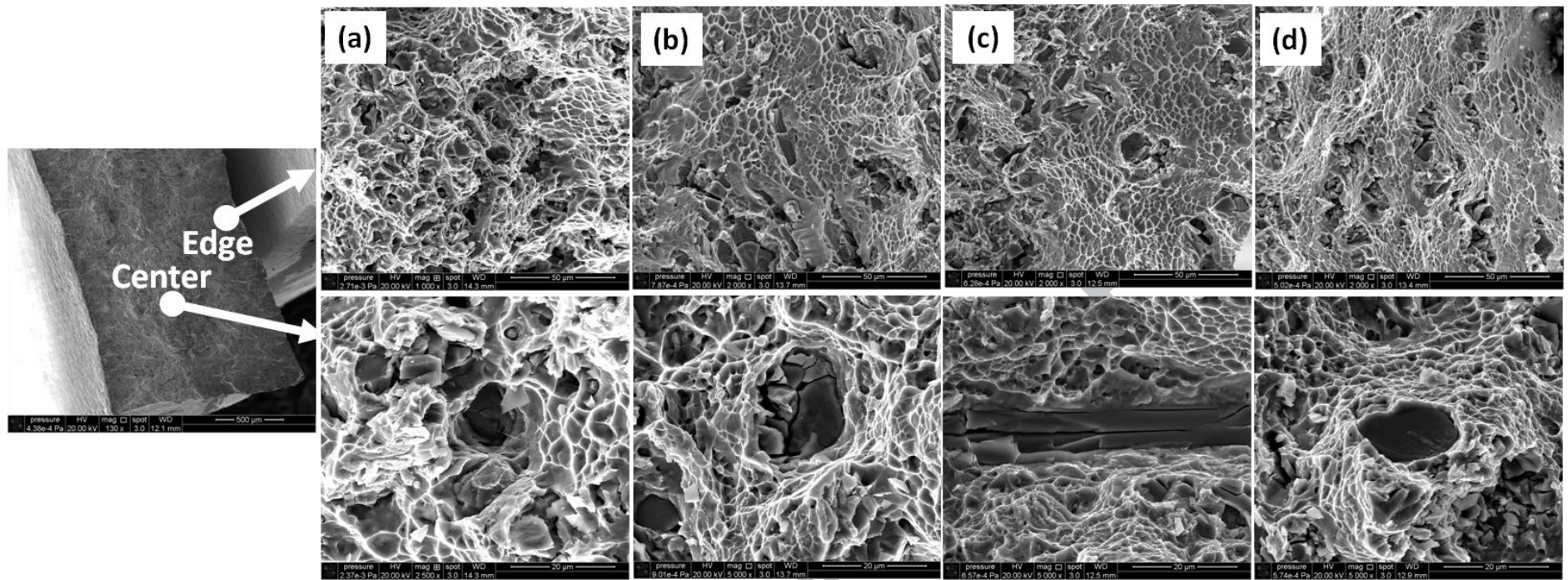
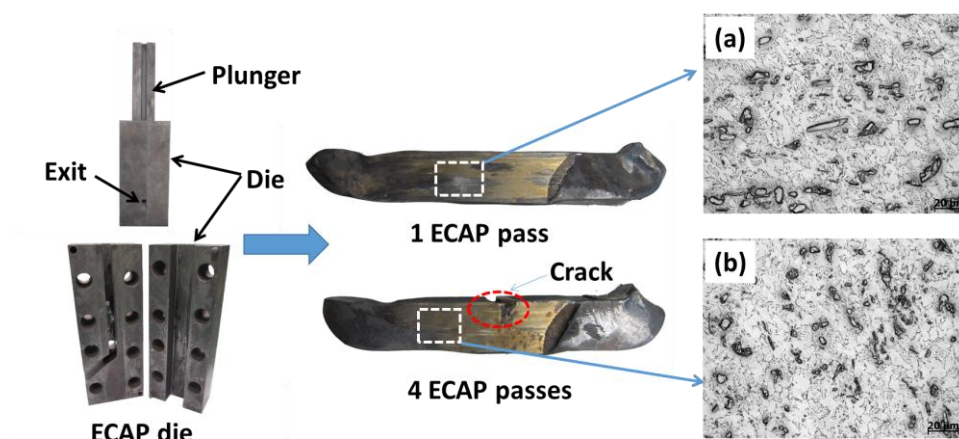


Fig. 9 Fracture surface of the ECAP-processed (TiB+TiC)/Ti6Al4V composite

(a) 1pass (b) 2 passes (c)3 passes (d)4 passes



### Abstract figure

Appearance of the titanium matrix composite billets after 1 and 4 ECAP passes at the same pressing speed. It showed that the surfaces of the samples are smooth and some small metal flash are found after 4 ECAP passes. Nevertheless, close inspections revealed that cracks appeared on the sample which was processed by 4 ECAP passes, and the microstructure showed the TiB and TiC reinforcements dispersed homogeneous in the composite.